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**FINAL REPORT**

**For DURIP Award # F49620-00-1-0245**

# X-ray Diffractometer for Texture and Residual Stress Studies of Advanced Materials

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## Abstract

Through AFOSR DURIP grant # **F49620-00-1-0245**, a four circle goniometer x-ray diffractometer outfitted with a high-temperature stage was purchased from Philips Analytical. The instrumentation facilitates residual stress measurements in highly textured materials up to 900°C. The instrument has significantly enhanced the AFOSR-funded research efforts of the PI on directionally solidified ceramic eutectics. Initial studies have measured the residual stresses in highly textured $Al_2O_3$-$ZrO_2(Y_2O_3)$ eutectics as a function of temperature. At room temperature, significant compressive stresses (~450 MPa) are present in $Al_2O_3$ with corresponding tensile stresses in $ZrO_2$. Through high-temperature studies, the stress-free temperature was found to be ~675°C.

## I. Objectives

The main objective of this DURIP grant was to procure a unique four-circle goniometer x-ray diffractometer (XRD) outfitted with a high-temperature domed furnace to monitor the stress state of the materials up to 900°C. This unique capability provides unprecedented insight into the stress evolution of these materials and allows us to determine a stress-free temperature of the material, which cannot be known a priori.

## II. Equipment Acquisition and Installation

The DURIP grant was awarded in 2000 and shortly thereafter the PI of the grant, Prof. Elizabeth Dickey, and one co-PI, Prof. Craig Grimes, decided to move to from the University of Kentucky to the Pennsylvania State University. Since the major research efforts that the instrumentation was intended to support were moving to Penn State, it was agreed upon by the University of Kentucky, Penn State and AFOSR that the money could be subcontracted to Prof. Dickey at Penn State. Therefore, a one-year no-cost extension was filed and approved and the budget was changed to reflect the subcontract.

After extensive investigation into available equipment, a purchase order was placed for the instrumentation from Penn State in April 2001. Philips Analytical received the order since they were the only company willing to outfit their four-circle goniometer XRD with a domed hot stage. Images of the instrument are shown in Fig. 1. Installation of the XRD took place in September 2001. The hot-stage installation was, however, significantly delayed due to integration problems by Philips. Ph.D. student, Colleen Frazer, was sent to Philips in the Netherlands in July and April of 2002 to do initial testing of the integrated system.

The hot stage was installed on the PSU XRD in October 2002. Since that time, we have been utilizing the instrument to measure residual stresses in directionally solidified eutectics, in support of AFOSR grant #F49620-02-1-0211 (Dr. Joan Fuller, program manager).

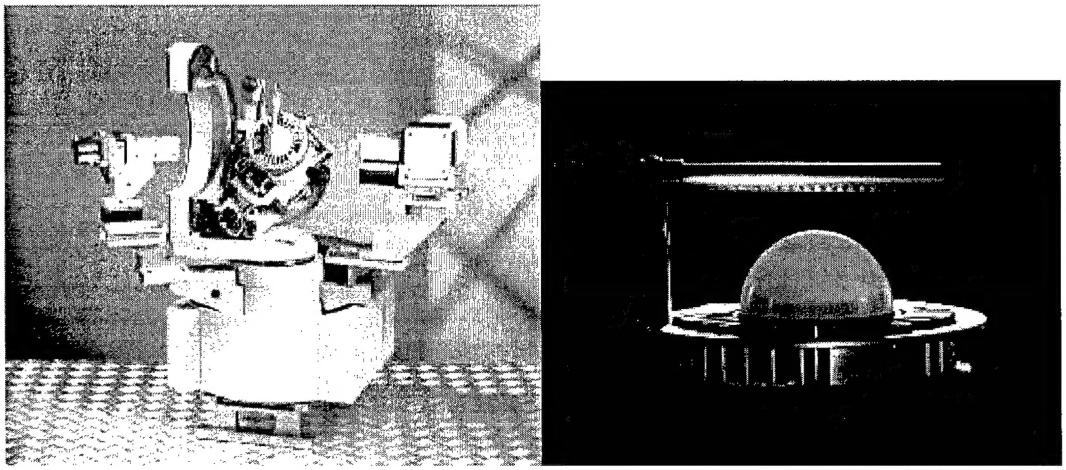

Fig. 1) four-circle goniometer Philips MRD x-ray outfitted with domed hot stage. b) close up of domed hot stage operated at 900°C.

## III. Application of Instrumentation: Measurement of Residual Stresses in Highly Textured Composites by X-ray Diffraction

Directionally-solidified oxide eutectics such as alumina-YAG and alumina-zirconia show promise as high-temperature structural materials because of their high temperature strength and creep resistance.[1-5] Since these materials are being designed for ultra-high temperature (1400°C) structural applications, it is imperative to understand the stability of the materials at high temperature and under thermal cycling. Compatibility constraints at the internal interfaces between the two constituent phases can lead to residual stresses upon thermal cycling and elastic interaction stresses under applied loads.[6] The magnitudes and distributions of these stresses have important ramifications for the mechanical behavior of the composites. In this AFOSR-supported research program we investigated thermal stresses in alumina—yttria-stabilized-zirconia (YSZ) directionally solidified eutectics (DSEs) from room temperature to 900°C. X-ray diffraction is employed to measure the strain tensors in each phase, which are subsequently converted to stress tensors. The research program is unique in that it provides the first insight into the high-temperature stress state of these materials.

Since DSEs are highly textured, it is important to first quantify the degree of texture in the materials via pole figure analysis. Fig. 1 shows pole figures from $Al_2O_3$ and $ZrO_2(Y_2O_3)$ in an a typical $Al_2O_3$-$ZrO_2$ DSE. Whereas the $Al_2O_3$ is nearly single crystalline with [0001] oriented along the growth axis, the $ZrO_2$ has multiple orientations, although it is highly (220) textured along the growth axis. This type of crystallographic texture was typical for all $Al_2O_3$-$ZrO_2$ DSEs studied, regardless of composition or growth parameters. This is in

contrast to YAG-$Al_2O_3$ DSEs whose crystallography is highly dependent on growth conditions.[7]

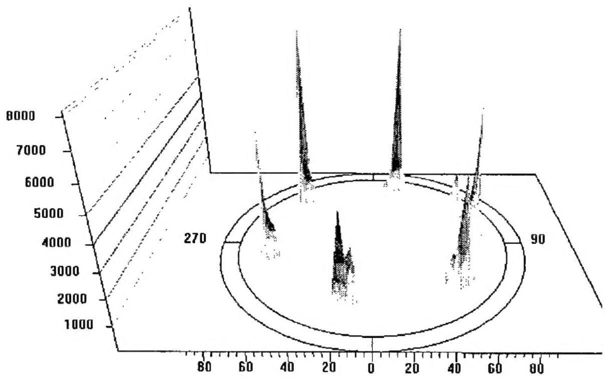


Figure 2a: (1123) pole figure of the $Al_2O_3$ phase in an $Al_2O_3$-$ZrO_2$ DSE. Since the phase is nearly single crystalline, a rotated single crystal stiffness tensor can be used.

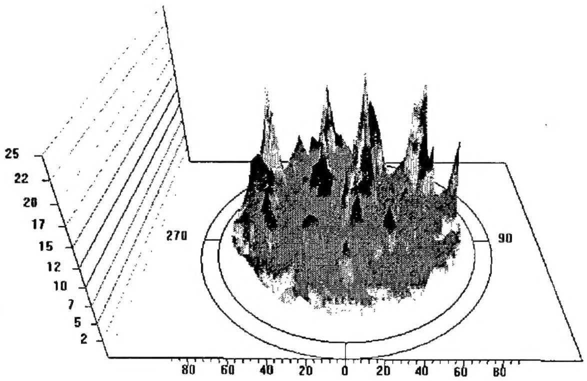


Figure 2b: (311) pole figure of the $ZrO_2$ phase in an $Al_2O_3$-$ZrO_2$ DSE. Since the phase is polycrystalline yet textured, a weighted stiffness tensor must be used.

Because the materials are highly textured, standard polycrystalline stress measurements using x-ray diffraction are not possible. Furthermore, because the materials are composites, the out-of-plane microstresses do not vanish within the sampling depth, so full triaxial stresses must be measured. The protocol for measuring stresses is outlined in two previous publications by the PI.[6,8] In general, at least six interplanar-spacing measurements are made along different crystallographic directions and the unstressed lattice parameter measured, so that it is possible to fit the six components of the strain tensor to the experimental data. Typically, the system is over-determined by making at least twelve measurements and using a fitting routine to determine the strain tensor and error matrix.

The final step in the analysis is to convert the strain tensors to stress tensors with the stiffness tensors. Since the alumina is nearly single crystalline, the single-crystal stiffness tensor rotated to the correct reference frame can be used. The $ZrO_2$ phase, however, has much weaker texture and it is necessary to weight the stiffness tensor using the orientation distribution function (ODF) as outlined below:

Applying this procedure to $Al_2O_3$-$ZrO_2$(6.6wt% $Y_2O_3$) DSEs, the following stress tensor was measured in $Al_2O_3$ at room temperature where $x_3$ is normal to the growth axis (and parallel to the c-axis of $Al_2O_3$):

$$\begin{vmatrix} -285 & -2 & -15 \\ & -327 & -7 \\ & & -354 \end{vmatrix} \quad +/- \quad \begin{vmatrix} 10 & 2 & 1 \\ & 10 & 4 \\ & & 6 \end{vmatrix} \quad \text{(MPa)}$$

These stresses are actually low, only ~ 65% of those predicted from finite element modeling, assuming the stress-free temperature to be the eutectic temperature of 1880°C. These low stresses suggest that enough mobility was present during part of the cooling stage to prevent some residual stress accumulation. To understand if stress mitigation processes dynamically occur upon thermal cycling, we annealed the specimen at 1600°C for 5 hours and slowly cooled the specimen. Room temperature stresses were then remeasured, but showed no appreciable change.

Analogous stress measurements were made at elevated temperatures utilizing the domed hot-stage outfitted on a four-circle goniometer x-ray diffractometer as shown in Fig. 1. Residual stress measurements were made at 400°C and 900°C. Loosely pressed powder specimens of Si (NIST standard) and $Al_2O_3$ were also measured at these temperatures for calibration purposes. The resulting stress tensors for $Al_2O_3$ are shown below:

$Al_2O_3$ (400°C):

$$\begin{vmatrix} -133 & 94 & -57 \\ & -26 & -0 \\ & & -116 \end{vmatrix} \pm \begin{vmatrix} 10 & 2 & 1 \\ & 13 & 4 \\ & & 7 \end{vmatrix} \text{ (MPa)}$$

$Al_2O_3$ (900°C):

$$\begin{vmatrix} 335 & 86 & -45 \\ & 427 & 0 \\ & & 409 \end{vmatrix} \pm \begin{vmatrix} 10 & 2 & 1 \\ & 13 & 4 \\ & & 7 \end{vmatrix} \text{ (MPa)}$$

As expected, the stresses decreased at 400°C to less that half of the room temperature values. At 900°C the $Al_2O_3$ went into tension indicating that the stress-free temperature is on the order of 500-600°C. So in service conditions of 1200-1400°C significant tensile stress will be present in the $Al_2O_3$.

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**Personnel Supported**

This equipment grant did not directly fund any personnel, but it did support the research activities of the following people, who were funded by AFOSR:

| | |
|---|---|
| Elizabeth C. Dickey | Associate Professor, Pennsylvania State University |
| Colleen S. Frazer | Graduate Student, University of Kentucky |
| C. Evan Jones | Undergraduate Student, Pennsylvania State University |
| Hongqi Deng | Graduate Student, Pennsylvania State University |

**Publications resulting from grant**

1. "High-Temperature Residual Stresses in $Al_2O_3$—$ZrO_2(Y_2O_3)$ Directionally Solidified Eutectics," C.S. Frazer and E.C. Dickey, in preparation for *Journal of the American Ceramic Society.*

2. "Residual Stresses in Directionally Solidified Oxide Eutectics" Ph.D. dissertation, University of Kentucky, in preparation.

**Patents and Inventions**

No patents or inventions resulted from this grant.